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TOBACCO SMOKE FILTER

This invention relates to tobacco smoke filters containing particulate sorbent.

Such use of sorbent particles to remove vapour phase (VP) components from tobacco smoke is well known. Cigarettes containing volatile flavourant (e.g. menthol) are also well known. However, prior attempts to use both volatile flavourant and particulate sorbent in a filter cigarette have been unsuccessful, it having proved impossible to provide a satisfactory level of flavour delivery whilst maintaining a satisfactory level of VP constituent removal by the particulate sorbent.

We have found that this problem can be overcome by a tobacco smoke filter containing activated carbon in which (1) pores of under 2 nm pore diameter (micropores) provide a pore volume of at most 0.3 cm^3/g (N_2); and (2) (a) pores of 2 to 50 nm pore diameter (mesopores) provide a pore volume of at least 0.25 cm^3/g (N_2) and/or (b) pores of 7 to 50 nm diameter (larger mesopores) provide a pore volume of at least 0.12 cm^3/g (Hg). An activated carbon without micropore volume has poor VP removal performance which is reduced yet further or nullified in the presence of volatile flavourant, and the indicated micro/meso pore

combinations are necessary to permit the required balance of flavour delivery and VP removal. Herein a pore volume expressed in cm^3/g (N_2) means said volume as measured by nitrogen porosimetry, using a Micromeritics Tristar 3000 for measurement of the nitrogen adsorption/desorption isotherms and characterising the pore size distribution via the BJH method on the desorption branch of the isotherm. A pore volume or surface area expressed in cm^3/g (Hg) or m^2/g (Hg) means said value as measured by mercury porosimetry using a contact angle of 140° and a surface tension value of 480 dynes/cm.

Accordingly the present invention provides a tobacco smoke filter containing activated carbon which carbon has a micropore volume provided by micropores of under 2 nm pore diameter, said micropore volume being up to 0.3 cm^3/g (N_2), and in which carbon mesopores of 2 to 50 nm pore diameter provide a mesopore volume of at least 0.25 cm^3/g (N_2); such a filter wherein at least 0.12 cm^3/g (Hg) of said mesopore volume is provided by mesopores of 7 to 50 nm pore diameter; and a tobacco smoke filter containing activated carbon which carbon has a micropore volume provided by micropores of under 2 nm pore diameter, said micropore volume being up to 0.3 cm^3/g (N_2), and in which carbon mesopores of 7 to 50 nm pore diameter provide a mesopore volume of at least 0.12 cm^3/g (Hg).

In the activated carbon used according to the invention pores of over 50 nm pore diameter (macropores) preferably provide a pore surface area of at least 5 m^2/g (Hg), most preferably of 6 or more m^2/g (Hg).

The designation of pores of less than 2 nm, 2 to 50 nm, and over 50 nm size (diameter) as micro-, meso- and macro-pores is in accord with accepted IUPAC terminology and definition.

The micropore volume provided by said micropores is preferably at most 0.26 cm^3/g (N_2), more preferably 0.15 cm^3/g (N_2) or less. The mesopore volume provided by said 2 to 50 nm mesopores may for example be about 0.3 cm^3/g (N_2) and is preferably over 0.4 or over 0.5 cm^3/g (N_2); the preferred range is thus from 0.3 to 0.5 or higher cm^3/g (N_2). The mesopore volume provided by the 7 to 50 nm larger mesopores is preferably 0.13 cm^3/g (Hg) or higher, and can be over 0.3 or over 0.5 cm^3/g (Hg); the preferred range is thus from 0.13 to 0.5 or higher cm^3/g (Hg).

We have most unexpectedly found that activated carbon of such carefully controlled micro/meso porosity - and preferably micro/meso/macro porosity - (a) shows a satisfactory level of adsorption of volatile flavourant such as menthol (not too little and not too much); (b) releases sufficient of the flavourant under smoking

conditions to deliver satisfactory taste; (c) shows good adsorption of VP components from tobacco smoke; and (d) retains a satisfactory (albeit reduced) level of this VP removal even in the presence of volatile flavourant such as menthol. This combination of properties has not heretofore been attainable.

Accordingly the invention also provides a tobacco smoke filter according to the invention incorporated in a filter cigarette containing volatile flavourant - e.g. menthol. Such a filter cigarette provides for the first time the combination of flavour delivery to give an acceptably flavoured taste with an acceptable reduction in delivery of VP smoke components.

The filter according to the invention may be of any design previously proposed for particulate sorbent-containing tobacco smoke filters. For example the carbon may be dispersed throughout a filter plug, carried on the tow or fibres or sheet material which is gathered to form the plug; it may instead be adhered to one or more threads which extend through the matrix of the filter plug or be adhered to the inner face of a wrapper around the filter plug; or it may form a bed sandwiched between a pair of plugs (e.g. of cellulose acetate tow) in a common wrapper. The carbon may be treated with the flavourant prior to filter production so that it acts as a carrier for the

flavourant and minimises migration of the flavourant during storage. Instead, the carbon could be used in a suitable filter in the unflavoured state, with the flavourant being added to another part of the filter and/or to the cigarette with which the filter is used and/or to the filter cigarette packaging. The flavourant might be carried on a wrapper around a filter plug or on one or more threads through a filter plug, and such plug may be the plug which also carries the activated carbon or a separate plug.

Filters according to the invention may additionally include one or more particulate sorbents other than the activated carbon required by the invention (e.g. silica gel, or a different carbon), mixed with the carbon required by the invention and/or separate from this.

The invention is illustrated by the following Examples, in which Examples B, C, D and H are according to the invention and the remainder are comparisons.

EXAMPLES

For each Example a sample of the respective activated carbon was dried and exposed to a menthol atmosphere in a desiccator at 55°C for 4 days, and the increase in weight was recorded. 'Triple granular' cigarette filters were then assembled, each containing 100 mg of the mentholated

carbon in a packed bed between two cellulose acetate filter segments. The filter cigarettes were smoked under ISO conditions (35 cm^3 puffs, each of two seconds duration, taken once per minute) and the menthol yields from the cigarettes were measured. The vapour phase of cigarette smoke was also collected and the percentage reduction of a selected number of vapour phase compounds measured; the mean reduction in these VP compounds, and the reduction obtained from an equivalent filter with 100 mg of the same carbon prior to exposure to menthol, were measured relative to an equivalent filter cigarette with no carbon.

The results are summarised in the following Table which gives the porosity parameters for the various carbons employed and the measured performances of the filters using them. Examples B, C, D and H used activated carbons according to the requirements of the invention, whilst the remainder did not. Comparison Example A used a standard coconut-based carbon as typically used in prior cigarette filters, whilst Comparison Examples E to G and I to M used other carbons whose micro/meso/macro porosity led to poor results. Comparison Example K showed good menthol uptake and yield, but with immeasurably low carbon micropore volume its VP removal performance was low and reduced to substantially zero in the presence of menthol. Comparison Examples I, J, L and M showed active VP removal

after mentholation but gave markedly inadequate menthol yield, whilst the remaining Comparison Examples (A and E to G) were markedly inadequate for both VP removal and menthol yield.

TABLE

EXAMPLE	A	B	C	D	E	F	G
Micropore Volume -(cm^3/g) (N_2)	0.46	0.26	0.11	0.12	0.52	0.33	0.23
2-50 nm Mesopore Volume -cm^3/g (N_2)	0.09	0.30	0.44	0.51	0.36	0.25	0.04
7-50nm Mesopore Volume -cm^3/g (Hg)	0.06	0.13	0.34	0.54	0.21	0.15	na *
Macropore Area -m^2/g (Hg)	1.9	6.4	6.9	12.2	1.4	4.9	na *
Menthol Uptake %	18.6	27.3	27.5	23	57.1	18.9	11.5
Menthol Yield (mg/cig)	0.03	0.73	0.44	0.72	0.07	0.06	0.15
Mean VP (unmentholated) (%)	53	85	45	61	85	45	47
Mean VP (mentholated) (%)	<5	24	24	36	<5	<5	<5

TABLE (Continued)

EXAMPLE	H	I	J	K	L	M
Micropore Volume -(cm^3/g) (N_2)	0.19	0.35	0.43	nm *	0.50	0.31
2-50 nm Mesopore Volume -cm^3/g (N_2)	0.29	1.05	0.92	0.49	1.10	0.28
7-50nm Mesopore Volume -cm^3/g (Hg)	0.13	0.20	0.29	na *	0.21	0.10
Macropore Area -m^2/g (Hg)	8.0	2.8	1.1	na *	2.5	3.7
Menthol Uptake %	20	34.6	34.9	39.7	39.1	20
Menthol Yield (mg/cig)	0.58	0.08	0.12	0.99	0.04	0.00
Mean VP (unmentholated) (%)	79	87	75	27	91	55
Mean VP (mentholated) (%)	25	46	45	0	30	35

* na - not ascertained

* nm - effectively zero - too small to measure